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Indian Standard

SPECIFICATION FOR POTASSIUM CHLORIDE, TECHNICAL

(Second Revision)

UDC 661·832·31

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR POTASSIUM CHLORIDE, TECHNICAL

(Second Revision)

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Indian Standard

SPECIFICATION FOR POTASSIUM CHLORIDE, TECHNICAL

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 25 January 1984, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1967. With a view to meeting the needs of the industry, the standard was revised in 1976, when two grades of potassium chloride were prescribed — grade 1 for electrolytic industry for manufacture of caustic potash and grade 2 for chemical industry in general. For Grade 1, optional requirements for trace metals like vanadium, chromium, titanium, molybdenum, copper, nickel and aluminium had been prescribed. In Grade 2, the limits for calcium and sulphate content had been modified. Perchloric acid method for the determination of potassium chloride content had been replaced by the tetraphenylboron and flame photometer methods.

0.3 The standard has been again revised incorporating a new method of calculation for the estimation of potassium chloride. The requirement for moisture content has been deleted from the table and has been prescribed under the general requirements, as the characteristics other than moisture have been expressed on dry basis.

0.4 Potassium chloride is used in the manufacture of caustic potash and potassium chlorate by electrolysis. It is also used in the manufacture of various other potash chemicals.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for potassium chloride, technical.

2. GRADES

2.1 There shall be two grades of the material, namely:

a) *Grade 1* — for use in the manufacture of caustic potash by electrolysis.

b) *Grade 2* — for use in chemical industry in general for the manufacture of potassium salts.

3. REQUIREMENTS

3.1 Description — The material shall be crystalline solid or powder, white, light grey or light pink in colour. It shall be free from visible contaminants like clay, grit and other extraneous adulterants and impurities.

3.2 Moisture Content — Grade 1 and Grade 2 material shall contain not more than 2·0 percent moisture, on as received basis when tested in accordance with the method prescribed in **A-3.**

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR POTASSIUM CHLORIDE, TECHNICAL

Sl No.	Characteristic	Requirement		Method of Test Ref to Cl No. in Appendix A
		Grade 1	Grade 2	
		(on Dry Basis)		
(1)	(2)	(3)	(4)	(5)
i)	Matter insoluble in water, percent by mass, *Max*	0·15	0·50	A-4
ii)	Sodium chloride (as NaCl), percent by mass, *Max*	1·00	1·5	A-5
iii)	Calcium (as Ca), percent by mass, *Max*	0·1	0·2	A-6
iv)	Magnesium (as Mg), percent by mass, *Max*	0·03	0·07	A-6
v)	Sulphates (as SO_4), percent by mass, *Max*	0·3	0·6	A-7
vi)	Iron (as Fe), parts per million, *Max*	20	20	A-8
vii)	Potassium chloride (as KCl), percent by mass, *Min*	98·5	97·0	A-9

3.4 For the manufacture of caustic potash by electrolytic process the material of Grade 1 shall comply with the following additional requirements when tested in accordance with the methods prescribed in **A-10** to **A-13**.

Characteristic	*Requirement*	*Method of Test*
Nitrates and ammonium salts (as NH_4), parts per million, *Max*	50	A-10
Vanadium + Chromium + Molybdenum, parts per million, *Max*	0·01	A-11
Aluminium (as Al), parts per million, *Max*	0·10	A-12
Other trace metals (titanium, copper, nickel), parts per million, *Max*	0·10	A-13

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in jute bags or as agreed to between the purchaser and the supplier.

4.2 Marking — The packages shall be securely closed and marked legibly and indelibly with the following information:

a) Name and grade of the material;

b) Mass of the material in the package;

c) Name of the manufacturer and his recognized trade-mark, if any; and

d) Lot or batch number in code or otherwise.

4.2.1 The packages may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their criteria for conformity shall be determined in accordance with the methods prescribed in Appendix B.

APPENDIX A

(*Clause* 3.3)

METHODS OF TEST FOR POTASSIUM CHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF SAMPLE

A-2.1 Crush 100 g of the sample to pass through 1-mm IS Sieve, dry in an air-oven at about 150°C for at least 4 hours. Cool in a desiccator and keep in a clean glass stoppered weighing bottle in a desiccator for subsequent tests.

A-2.2 Weigh 50 g of the sample (**A-2.1**), dissolve in 200 ml of water and add 25 ml of concentrated hydrochloric acid. Heat to boiling, cool and filter into a 500-ml measuring flask. Wash the paper with water till it is free from chloride and make up the volume to 500 ml. Use this solution for all subsequent tests except for trace metals.

A-2.2.1 For trace metal analysis, dissolve completely 3 200 g of salt sample in water, acidify with 100 ml of concentrated hydrochloric acid and stir well to dissolve all the soluble matter. Filter and make up to 10 litres.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 20 g of the material as received in the weighing bottle (about 30-ml capacity), preferably wide mouth squat type, previously dried and weighed. Dry in an oven at 140-150°C for at least 4 hours. Cool in a desiccator and weigh. Repeat drying, cooling and weighing until constant mass is obtained.

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*Specification for water for general laboratory use (*second revision*).

A-3.2 Calculation

$$\text{Moisture, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = initial mass in g of the material taken for the test, and
M_2 = final mass in g of the material after drying.

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Procedure — Dissolve 20 g of the sample (**A-2.1**), accurately weighed in about 200 ml of water. Filter through sintered glass crucible (G No. 4) and wash the residue with water till the filtrate gives no opalescence with silver nitrate solution. Preserve this solution after making up the volume to 1 litre for subsequent tests. Dry the sintered glass crucible with the residue to constant mass at 105 to 110°C.

A-4.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and
M_2 = mass in g of sample taken for the test.

A-5. DETERMINATION OF SODIUM CHLORIDE CONTENT

A-5.1 Apparatus

A-5.1.1 *Simple Flame Photometer* — equipped with interference filter.

A-5.2 Reagents

A-5.2.1 *Standard Sodium Chloride Solution* — Weigh accurately 5 g of sodium chloride, dissolve it in water, transfer to 1 litre measuring flask and make up the volume. The standard solution contains 0·5 g of sodium chloride in 100 ml.

A-5.2.2 *Standard Solution of Potassium Chloride for Buffer* — Recrystallize potassium chloride and dry at 110°C. Dissolve 10 g of this recrystallized potassium chloride in 100 ml of water.

A-5.2.3 *Sample Solution* — Accurately weigh 1·0 g of the sample (**A-2.1**) and dissolve in 1 000 ml of water in a measuring flask.

A-5.2.4 *Calibration Graph* — Since sodium chloride is an impurity in potassium chloride which may interfere in the determination of sodium chloride, bracketing technique or internal calibration of sodium chloride in presence of potassium chloride is adopted. Buffer standard solution is used for calibration.

A-5.2.5 *Buffer Standard Sodium Chloride Solution* — Bracketed with potassium chloride (*see* **A-5.3.1**).

A-5.3 Procedure

A-5.3.1 Pipette out different aliquots 1, 2, 4, 5, 6, 8 and 10 ml of standard sodium chloride solution **A-5.2.1** into a number of 100-ml measuring flasks. Each flask respectively contains 5, 10, 25, 30, 40, 50 mg of sodium chloride. Pipette out 10 ml of standard potassium chloride solution (1·0 g KCl) to each of the flasks and make up the volume to 100 ml. This is buffered solution with potassium chloride. Use this solution for calibration graph.

A-5.3.2 Use a flame photometer equipped with atomizer and burner; optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-5.3.3 Insert the sodium filter corresponding to wavelength 589 mm; light the burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0·5 to 0·7 kgf/cm^2 and maintain the above air pressure constant such that the flame becomes non-luminous on turning the gas control knob. First spray water and adjust the pointer to zero on galvanometer scale by zero adjustment knob. Then spray the standard sodium chloride (*see* **A-5.2.1**) and adjust the deflection to maximum 100 by using sensitivity control knob. Again spray water to see that pointer comes to zero; then spray standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with same adjustment during both the operations. Now reading zero with water and with the same adjustment 100 with the standard solution indicates that the instrument is ready for measurement.

A-5.3.4 Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **A-5.3.1** and obtain a calibration graph.

A-5.3.5 Use the buffer solution containing 0·05 g of sodium chloride per 100 ml for obtaining maximum deflection of 100 divisions of galvanometer. Sodium chloride range in the calibration graph is 0·005 to 0·05 sodium chloride in 100 ml and the impurity of sodium chloride exists in the same proportion with potassium chloride.

A-5.3.6 After washing with water, spray the sample solution (*see* **A-5.2.3**) containing approximately 0·1 g potassium chloride per 100 ml and obtain the galvanometer reading. From the graph, read the corresponding concentration of sodium chloride in 100 ml (say A).

A-5.4 Calculation

$$\text{Sodium chloride, percent by mass} = \frac{A}{M} \times 100$$

where

A = concentration of sodium chloride in g per 100 ml of the sample solution, and

M = mass in g of the sample taken for preparing 100 ml of sample solution.

A-6. DETERMINATION OF CALCIUM AND MAGNESIUM

A-6.1 Reagents

A-6.1.1 *Standard Calcium Solution* — Weigh 1·0 g of calcium carbonate dried at 120°C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask. One millilitre of the solution is equivalent to 0·400 8 mg of calcium (as Ca).

A-6.1.2 *Standard EDTA Solution* — Dissolve 3·72 g of disodium ethylene diamine tetraacetate dihydrate in water and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against standard calcium solution following the procedure given in **A-6.2**.

A-6.1.3 *Eriochrome Black T Indicator Solution* — Dissolve 0·1 g of the dye in 20 ml of rectified spirit (*see* IS : 323-1959*). This solution shall be prepared fresh every week.

A-6.1.4 *Dilute Sodium Hydroxide Solution* — approximately 10 percent.

A-6.1.5 *Patton and Reeder's (P & R) Indicator* — Mix 0·1 g of 2-hydroxy-1-(2-hydroxy-4-sulpho-naphthalxo)-3-naphthoic acid with 10 g of potassium chloride and grind into a fine mixture in a glass mortar, preserve in a well stoppered dry amber coloured glass bottle.

A-6.1.6 *Calcein Indicator* — Grind thoroughly 0·1 g of calcein and 0·06 g of thymolphthalein with 10 g of potassium chloride.

A-6.1.7 *Ammonium Chloride-Ammonium Hydroxide Buffer Solution* — Dissolve 67·5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0·90) and 250 ml of water. Also dissolve separately a

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*Specification for rectified spirit (*revised*).

mixture of 0·931 g of disodium ethylene diamine tetraacetate dihydrate and 0·616 g of magnesium sulphate ($MgSO_4 \cdot 7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to 1 litre.

NOTE — Five millilitres of buffer solution added to 50 ml of water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

A-6.2 Procedure

A-6.2.1 *Standardization of EDTA Solution* — Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against standard EDTA solution to a pure blue end-point.

A-6.2.2 Titrate 25 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0·1 ml) from the reading obtained in **A-6.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

A-6.2.3 Transfer exactly 100 ml of the solution preserved in **A-4.1** into a 250-ml conical flask. Add 10 ml of the ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine-red colour of the solution changes to pure blue end-point. Note the EDTA solution used in the titration (V_1).

A-6.2.4 Transfer exactly 100 ml of the solution preserved in **A-4.1** into 250-ml concial flask, add 5 ml of sodium hydroxide solution and stir well. Further, add 0·2 g of P & R indicator (or 100 mg of calcein mixed indicator) and titrate against standard EDTA solution till wine-red colour of the solution changes to purple violet end-point with P & R indicator (or green to purple with calcein indicator). Note the volume of the standard EDTA solution used in the titration (V_2).

A-6.3 Calculation

Calcium (as Ca), percent by mass (on dry basis) $= \frac{A V_2}{M}$

Magnesium (as Mg), percent by mass (on dry basis) $= 0.606\,8 \frac{(V_1 - V_2)}{M}$

where

A = calcium equivalent in mg of 1 ml of EDTA solution determined in **A-6.2.1**,

V_1 = volume in ml of standard EDTA solution used in **A-6.2.3**,

V_2 = volume in ml of standard EDTA solution used in **A-6.2.4** (for Ca only), and

M = mass in g of the dried sample taken for the test in **A-4.1**.

A-7. DETERMINATION OF SULPHATE

A-7.1 Gravimetric Method

A-7.1.1 *Reagents*

A-7.1.1.1 *Dilute hydrochloric acid* — approximately 4 N.

A-7.1.1.2 *Barium chloride solution* — approximately 10 percent.

A-7.1.2 *Procedure* — Dissolve about 10 g of dried sample preserved in **A-2.1** in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Add one drop of methyl orange and 10 ml of dilute hydrochloric acid or more till it is pink and then boil. Add to the boiling solution, 10 to 12 ml of barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 minutes to obtain a granular precipitate. Allow to stand for 4 hours and filter through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash the precipitate till free from chloride and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and determine as barium sulphate.

A-7.1.3 *Calculation*

$$\text{Sulphate (as } SO_4 \text{), percent by mass} = 41{\cdot}13 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of barium sulphate, and

M_2 = mass in g of dried sample taken for the test.

A-7.2 Volumetric Method

A-7.2.1 *Reagents*

A-7.2.1.1 *Standard barium chloride solution* — 0·05 N. Dissolve 6·108 g of barium chloride dihydrate ($BaCl_2{\cdot}2H_2O$) in water and make up to one litre.

A-7.2.1.2 *Dilute hydrochloric acid* — approximately 1 N.

A-7.2.1.3 *Standard EDTA solution* — Weigh 3·72 g of disodium ethylenediaminetetraacetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against standard barium chloride solution (**A-7.2.1.1**) following the procedure given in **A-7.2.2**. One millilitre of standard EDTA solution is equal to 0·001 374 g of barium (Ba) or 0·000 96 g of sulphate (SO_4).

A-7.2.1.4 *Eriochrome black T indicator solution* — same as in **A-6.1.3**.

A-7.2.1.5 *Ammonium chloride-ammonium hydroxide buffer solution* — Same as in **A-6.1.7**.

A-7.2.2 *Procedure*

A-7.2.2.1 Pipette out 20 ml of standard barium chloride solution (**A-7.2.1.1**) into a conical flask, add 2 drops of hydrochloric acid and 10 ml of ammonium chloride—ammonium hydroxide buffer solution. Dilute it with water to about 50 ml, add five drops of arichrome black T indicator solution and titrate against standard EDTA solution to pure blue end point. Note the titre value (say A).

A-7.2.2.2 Pipette out 100 ml of the solution preserved in **A-2.2** and add two drops of hydrochloric acid and heat to gentle boiling. To the hot solution, add 20 ml of standard barium chloride solution. Boil gently for about 5 minutes and then cool to room temperature. The solution will be neutral or slightly acidic when tested with a litmus paper. Add 10 ml of ammonium chloride-ammonium hydroxide buffer solution and five drops of eriochrome black T indicator solution. Titrate against standard EDTA solution to pure blue end point. Note the titre value (say B).

A-7.2.3 *Calculation*

$$\text{Sulphate (as } SO_4 \text{), percent by mass (on dry basis)} = 0{\cdot}96\,\frac{A + V_1 - B}{M}$$

where

A = volume in ml of standard EDTA solution used in **A-7.2.2.1**,

V_1 = volume in ml of standard EDTA solution used in **A-6.2.3** (total Ca + Mg),

B = volume in ml of standard EDTA solution used in titration in **A-7.2.2.2**, and

M = mass in g of the dried sample taken for the test in **A-2.2**.

A-8. TEST FOR IRON

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50-ml capacity.

A-8.2 Reagents

A-8.2.1 *Dilute Hydrochloric Acid* — approximately 5 N, free from iron.

A-8.2.2 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-8.2.3 *Potassium Thiocyanate Solution* — 5 percent (*m/v*).

A-8.2.4 *Dilute Sulphuric Acid* — approximately 5 N.

A-8.2.5 *Standard Iron Solution* — Dissolve 2·81 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$] in 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Transfer 10 ml of the solution to 1 000-ml volumetric flask and dilute again to mark. One millilitre of this solution is equivalent to 0·004 mg of iron (as Fe).

A-8.3 Procedure — Weigh accurately about 2 g of the material and dissolve it in about 20 ml of water. Add about 5 ml of hydrochloric acid in order to make the solution and 2 ml of nitric acid and boil to oxidize the iron salts. Cool and transfer to a Nessler cylinder, add 5 ml of potassium thiocyanate solution, dilute to the mark with water and stir well. Into a second Nessler cylinder, add 5 ml of hydrochloric acid, 2 ml of nitric acid, 10 ml of standard iron solution and 5 ml of potassium thiocyanate solution. Dilute to the mark and stir well. Compare the colour of the solution in the two cylinders.

A-8.3.1 The limit prescribed in Table 1 shall be taken to have not been exceeded if the intensity of colour produced with the sample is not greater than that produced by the standard iron solution.

A-9. DETERMINATION OF POTASSIUM CHLORIDE CONTENT

A-9.0 Outline of the Method — Potassium chloride is determined by subtracting the values of sum of the chlorides due to calcium, magnesium and sodium from total chloride.

A-9.1 Estimation of Total Chloride

A-9.1.1 Reagents

A-9.1.1.1 *Standard silver nitrate solution* — 0·1 N.

A-9.1.1.2 *Potassium chromate indicator* — 5 percent in water (*m/v*).

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*Specification for nitric acid (*second revision*).

A-9.1.2 *Procedure* — Weigh accurately about 1·0 g of the material (**A-2.1**), dissolve in 50 ml of water and dilute to 100 ml in volumetric flask. Take 10 ml in a flask and titrate with 0·1 N silver nitrate, using 1 ml of potassium chromate as indicator till a reddish brown tinge persists after brisk shaking. Carry out a blank determination using water.

A-9.1.3 *Calculation*

$$\text{Total chloride (including KCl, MgCl}_2\text{, CaCl}_2\text{ and NaCl), percent by mass} = \frac{V \times 0{\cdot}354\,6}{M}$$

where

V = volume in ml of standard silver nitrate solution used, and

M = mass in g of the sample present in the aliquot.

A-9.1.3.1 Residual chloride is the chloride obtained by deducting the chloride attributed to the other chlorides, such as magnesium chloride, calcium chloride (as described in **A-9.1.3.2**) and sodium chloride as described in **A-5.4** from the total chloride determined in **A-9.1.3.**

A-9.1.3.2 Express the soluble carbonate as calcium carbonate (marine salt), or as sodium carbonate (Rajasthan salt, in absence of calcium and magnesium). If there is excess of calcium over calcium carbonate, combine all the calcium with sulphate radical and express the result as percentage of calcium sulphate. If there is excess of the sulphate over what is required for calcium, combine the excess with magnesium and express the result as percentage of magnesium sulphate. Calculate the balance of magnesium as magnesium chloride and deduct the chlorides corresponding to it from the chloride content. Also deduct from the chloride content the amount of chloride corresponding to the sodium content and express it as percentage of sodium chloride. Calculate the residual chloride as percentage of potassium chloride.

A-9.1.3.3 Calculation

$$\text{Chloride due to KCl} = \text{Total chloride} - [(\text{Chloride due to NaCl}) + (\text{Chloride due to MgCl}_2)]$$

A-10. TEST FOR NITRATES AND AMMONIUM SALTS

A-10.0 Outline of the Method — The nitrates in the sample are reduced to ammonium salts by digestion with caustic soda and aluminium wire. The ammonia is distilled over and estimated by Nesslers reagent.

A-10.1 Reagents

A-10.1.1 *Sodium Hydroxide Solution* — 10 percent (*m/v*).

A-10.1.2 *Dilute Hydrochloric Acid* — 0·1 N (approximately).

A-10.1.3 *Aluminium wire*

A-10.1.4 *Nessler Reagent* — Dissolve 10 g potassium iodide in 10 ml of ammonia free water, and add to it slowly, while stirring saturated mercuric chloride solution, until a slight permanent precipitate is formed. Add 30 g potassium hydroxide and when it has dissolved add 1 ml of mercuric chloride solution and dilute to 200 ml with ammonia free water. Allow to settle overnight and decant the clear solution and store for further use.

A-10.1.5 *Standard Ammonium Chloride Solution* — Dissolve 0·296 g ammonium chloride in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. 1 ml of the diluted solution is equivalent to 0·01 mg of ammonium radical (NH_4).

A-10.2 Apparatus

A-10.2.1 *Nessler Cylinder* — 50 ml capacity.

A-10.3 Procedure — Dissolve 1·0 g of the material in 60 ml water in a flask connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 ml dilute hydrochloric acid. Add to the solution in the flask 20 ml of freshly boiled sodium hydroxide solution and about 0·5 g of aluminium wire, in small pieces. Allow to stand for an hour and distill over 35 ml. Transfer the distillate into a Nessler cylinder, add 2 ml of Nessler's reagent. Carry out a control test in another Nessler cylinder in the same manner, using ammonia free water and 5 ml standard ammonium chloride solution in place of the material.

A-10.3.1 The limit prescribed in Table 1 shall be taken to have not been exceed if the intensity of colour produced with the sample is not greater than that produced in the control test.

A-11. DETERMINATION OF VANADIUM, CHROMIUM AND MOLYBDENUM

A-11.0 Vanadium, chromium and molybdenum are first individually estimated as given in **11.1**, **11.2** and **11.3**, and the contents summed up to give their total contents (*see* **11.4**).

A-11.1 Estimation of Vanadium

A-11.1.0 *Principle* — Vanadium is extracted from a large quantity of brine with 8-hydroxyquinoline, and subsequently determined colorimetrically as phosphotungstate.

A-11.1.1 *Reagents*

A-11.1.1.1 *8-Hydroxyquinoline reagent* — Dissolve 4 g of the reagent in 100 ml of 10 percent acetic acid.

A-11.1.1.2 *Sodium acetate* — 5 percent.

A-11.1.1.3 *Chloroform — See* IS : 5296-1979*.

A-11.1.1.4 *Anhydrous sodium sulphate.*

A-11.1.1.5 *Sodium carbonate — See* IS : 296-1974†.

A-11.1.1.6 *Phosphoric acid* — 1 : 2 (*v/v*).

A-11.1.1.7 *Sodium tungstate solution* — 0·5 N. Dissolve 16·5 g of sodium tungstate in 100 ml of water.

A-11.1.1.8 *Standard vanadium solution* — Ignite pure vanadium pentoxide at 500°C for an hour and cool it in a desiccator. Dissolve 1·7850 g of this in a slight excess of sodium carbonate solution. Neutralize with sulphuric acid, add a few drops in excess and finally dilute to 1 000 ml. One millilitre of this solution is equivalent to 1·0 mg of vanadium. Further, dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 10 μg of vanadium.

A-11.1.2 *Procedure* — Take 4 litres of the sample solution (**A-2.2.1**). Adjust the *p*H to 4·5 to 5·5 by the addition of 50 percent sodium hydroxide solution or concentrated sulphuric acid. Add 80 ml of sodium acetate solution and 16 ml of 8-hydroxyquinoline reagent. Mix the contents thoroughly, check the *p*H and maintain it again to 4·5 to 5·5.

Take 1 litre at a time of the above solution in a separating funnel and extract the 8-hydroxyquinoline complex using 20 ml portions of chloroform. Combine the extracts and remove traces of water by adding anhydrous sodium sulphate. Transfer the chloroform extract to a platinum dish and evaporate chloroform at very low heat keeping the dish inside a well ventilated fume chamber. The solution has a tendency to creep up the sides of the dish and if necessary, carefully heat the sides of the dish with a burner.

When dry, place the dish in a muffle furnace at about 800 to 900°C and burn off all the organic matter. Add 5 g of anhydrous sodium carbonate and fuse carefully at a temperature of 900-1 000°C, occasionally swirling the dish to cover the sides also. After about 10 to 15 minutes, cool the dish, fill up two-thirds with water and warm gently to dissolve the melt. Transfer to a small beaker and filter through a filter paper (Whatman No. 42 or equivalent). Neutralize the filtrate with concentrated sulphuric acid to *p*H 7.

For blank, take 5 g of sodium carbonate in a beaker, dissolve in a small quantity of water and neutralize with concentrated sulphuric acid to *p*H 7. Add to both beakers enough sulphuric acid to make 0·5 N solution,

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*Specification for chloroform, pure and technical (*first revision*).

†Specification for sodium carbonate, anhydrous (*second revision*).

further add 5 ml of phosphoric acid and 2·5 ml of sodium tungstate solution. Keep the beakers on a hot-plate and boil gently for 2 minutes. Cool and make up the volume to 50 ml with water. A pale-yellow colour of the phospho-tungstate would develop in the sample. Find out the percentage transmittance of the solution taking the blank for 100 percent transmittance on the photoelectric colorimeter using a cell of 5 cm light path and a blue 400 nm filter. Find out the corresponding optical density and from the standard graph (*see* **A-11.1.3**), calculate the equivalent vanadium content.

A-11.1.3 *Preparation of Standard Graph* — Take varying volumes of standard vanadium solution in 100-ml beakers so as to obtain 5, 10, 25, 50, 75 and 100 μg of vanadium. Dilute to about 40 ml and make acidic with concentrated sulphuric acid to approximately 0·5 N. Add 5 ml of phosphoric acid and 2·5 ml of sodium tungstates olution to each beaker. Boil for 2 minutes on a hot-plate, cool and dilute to 80 ml. Also take a blank using 40 ml of water. Find out the percentage transmittance on a photoelectric colorimeter using blue 400 mm filter. Find out the corresponding optical densities and plot the vanadium concentrations against the corresponding optical densitities.

A-11.1.4 *Calculation*

$$\text{Vanadium content in the solution, parts per million} = \frac{a}{4\,000}$$

$$\text{Vanadium content } (X_1), \text{ parts per million in the sample} = 3{\cdot}125 \times \frac{a}{4\,000}$$

a = mass in μg of vanadium contained in 4 litres of the sample.

A-11.2 Estimation of Chromium

A-11.2.0 *Principle* — Chromium (hexavalent form) is reduced to chromium (trivalent form) by boiling with potassium iodide and sodium sulphite solution which is then co-precipitated with aluminium hydroxide. Aluminium hydroxide is dissolved in 1 : 1 sulphuric acid solution and chromium is oxidized to the hexavalent form by bromine water. It is then determined colorimetrically by reacting with diphenyl carbazide reagent which gives a purple colour with chromium.

A-11.2.1 *Reagents*

A-11.2.1.1 *Sulphuric acid solution* — 1 : 1 (*v/v*) and 1 : 9 (*v/v*).

A-11.2.1.2 *Phosphoric acid solution* — 1 : 5 (*v/v*).

A-11.2.1.3 *Aluminium solution* — Dissolve 1·0 g of aluminium in pure hydrochloric acid and dilute to 1 litre.

A-11.2.1.4 *Diphenyl carbazide reagent* — Dissolve 0·25 g of diphenyl carbazide and 0·4 g of phthalic anhydride in 100 ml of ethanol. The solution is stable for one month.

A-11.2.1.5 *Bromine water*

A-11.2.1.6 *Potassium iodide solution* — 10 percent.

A-11.2.1.7 *Sodium hydroxide solution* — 30 percent.

A-11.2.1.8 *Neutral sodium sulphite*

A-11.2.1.9 *Standard chromium solution* — Weigh 0·283 g of dried potassium dichromate, dissolve in a little water and dilute to 1 litre. One millilitre of this solution is equivalent to 0·1 mg of chromium (as Cr). Further, dilute 10 ml of the above solution to 1 litre. One millilitre of the diluted solution is equivalent to 1 μg of chromium.

A-11.2.2 *Procedure* — Take 2 litres of the sample solution (**A-2.2.1**) in a 4-litre beaker, add 40 ml of potassium iodide solution, 40 ml of sulphuric acid (1 : 1) and 200 ml of water. Boil for about 10 minutes and add sodium sulphite, in small portions, to decolorize the solution. In case the solution turns brown on further boiling, continue adding sodium sulphite till the decolorization is stable and the brown colour of the iodine does not reappear on boiling. Add 30 ml of aluminium solution and stir well. Further, add sodium hydroxide solution dropwise, stir the contents well and adjust the *p*H to 6·0 to 6·5. Allow to settle to complete precipitation. Siphon off the clear solution and collect the aluminium precipitate in a small beaker. Filter through a sintered glass crucible and wash once with hot water. Dissolve the precipitate by pouring 60 ml of boiling sulphuric acid solution (1 : 9) into the crucible, apply suction after 2 to 3 minutes and again wash with hot water. Collect the filtrate separately in a 250-ml beaker. Add 4 ml of bromine water and stir well. Decolorize the yellow colour with a few drops of sodium hydroxide solution and add 1 ml in excess. Boil with 1 : 1 sulphuric acid until the brownish colour appear. Add 2 ml of the acid in excess and boil again till the brownish colour disappears. Cool and add 1 ml of phosphoric acid. Make up to 100 ml and divide into two parts. Keep one as a blank and to the other add 1 ml of diphenyl carbazide solution. Make up both solutions to 100 ml. Wait for 10 to 15 minutes for the colour to develop fully and then find out the percentage transmittance on a photoelectric colorimeter using 530 mm green filter. Calculate the corresponding optical density. From the standard graph (*see* **A-11.2.3**) find out the corresponding concentration of chromium.

A-11.2.3 Take varying volumes of standard chromium solution so as to give 5, 10, 15, 20 and 25 µg of chromium. Add 15 ml of aluminium solution to each, dilute to about 50 ml, then add 30 ml of sulphuric acid solution and 2 ml of bromine water. Further, add sodium hydroxide solution dropwise till the colour disappears and 2 drops in excess. Boil for 5 minutes and cautiously add 1 : 1 sulphuric acid solution till the bromine colour reappears and add 0·5 ml of the acid in excess. Boil for about 20 minutes until bromine colour disappears. Check for bromine, cool and add 1 ml of diphynyl carbazide solution and make up the volume to 100 ml. Let it stand for 15 minutes and then take the reading on a photoelectric colorimeter using green 530 nm filter and a glass cell with 5 cm light path. Find out the corresponding optical density. For adjusting 100 percent transmittance, use 50 ml water and proceed in the same manner as above. Plot the optical densities against the corresponding chromium concentrations.

A-11.2.4 *Calculation*

Chromium content in the sample solution, parts per million $= \frac{a}{1\,000}$

Chromium (X_2), parts per million in the sample $= 3{\cdot}125 \times \frac{a}{1\,000}$

where

a = mass in µg of chromium contained in 1 litre of the sample.

A-11.3 Estimation of Molybdenum

A-11.3.0 *Principle* — Molybdenum is extracted as the 8-hydroxyquinoline complex and then converted into the sulphate which is further reacted with potassium thiocyanate in the presence of a strong reducing agent like stannous chloride to give a cherry red colour. The colour is extracted with an organic solvent and the intensity is measured on a photoelectric colorimeter.

A-11.3.1 *Reagents*

A-11.3.1.1 *Anhydrous potassium pyrosulphate*

A-11.3.1.2 *Sulphuric acid solution* — 10 percent (*v/v*).

A-11.3.1.3 *Ferrous ammonium sulphate solution* — 1 percent.

A-11.3.1.4 *Potassinm thiocyanate solution* — 5 percent.

A-11.3.1.5 *Stannous chloride solution* — 10 percent. Dissolve 10 g of stannous chloride in 20 ml of 1 : 1 hydrochloric acid and dilute to 100 ml. Filter if not clear.

A-11.3.1.6 *Mixture of isoamyl alcohol and carbon tetrachloride* — 1 : 1.

A-11.3.1.7 *Sodium molybdate*

A-11.3.1.8 *Standard molybdenum solution* — Dissolve 0·282 2 g of sodium molybdate in 1 000 ml of water. Further, dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 1 μg of molybdenum.

A-11.3.2 *Procedure* — Extract 2 litres of the sample solution (**A-2.2.1**) with 8-hydroxyquinoline by the method described in **A-11.1.2**. Evaporate chloroform completely in a well ventilated fume chamber and ignite at low temperature. Add 5 g of anhydrous potassium pyrosulphate and fuse in a muffle furnace at 850°C swirling the dish at intervals to spread the melt along the sides of the dish. Add sufficient quantity of sulphuric acid solution to dissolve the melt and transfer to a separating funnel which has been dried in an oven. Add 1 ml of ferrous ammonium sulphate solution, 10 ml of potassium thiocyanate solution and mix thoroughly. Add 10 ml of stannous chloride solution and make up the solution to about 50 ml.

Shake the contents with 20 ml portions of isoamyl alcohol-carbon tetrachloride mixture, collect the alcoholic layer into a 50-ml volumetric flask, and make up the volume to 50 ml. The extract shall be clear. If turbid, keep the flask in hot water for a few seconds. For blank, take 100 ml water, treat with the reagents as done for the sample and extract with 1 : 1 mixture of isoamyl alcohol and carbon tetrachloride. Adjust 100 percent transmittance with blank on the photoelectric colorimeter, using a 5-cm light path cell and 460 nm green filter. From the standard graph (*see* **A-11.3.3**) find out the corresponding molybdenum concentration.

A-11.3.3 *Preparation of Standard Graph* — Take different aliquots of the standard molybdenum solution so as to obtain 5, 10, 15, 20, and 25 μg of molybdenum. Add 5 ml of sulphuric acid solution and dilute to about 40 ml. Add 1 ml of ferrous ammonium sulphate solution, 10 ml of potassium thiocyanate solution and 10 ml of stannous chloride solution. Transfer to a separating funnel and extract with two 20-ml portions of isoamyl alcohol-carbon tetrachloride mixture. Combine extracts and make up to 50 ml with the same mixture. Find out the percentage transmittance using a photoelectric colorimeter with green 460 nm filter and a glass cell of 5-cm light path and find out the corresponding optical density. Plot the optical densities against the molybdenum concentration on a graph paper.

A-11.3.4 *Calculation*

$$\text{Molybdenum content in the solution, parts per million} = \frac{a}{2\,000}$$

$$\text{Molybdenum content } (X_3) \text{ in the sample, parts per million} = 3{\cdot}125 \times \frac{a}{2\,000}$$

where

a = mass in μg of molybdenum contained in 2 litres of the sample.

A-11.4 Total Vanadium, Chromium and Molybdenum

A-11.4.1 *Calculation*

$$(\text{Vanadium} + \text{Chromium} + \text{Molybdenum}), \text{ parts per million, in the sample} = X_1 + X_2 + X_3$$

A-12. DETERMINATION OF ALUMINIUM

A-12.0 Principle — Aluminium forms red coloured lake with aluminon reagent. It can then be colorimetrically estimated.

A-12.1 Reagents

A-12.1.1 *Buffer Solution* — Dissolve 154 g of ammonium acetate and 6 ml of acetic acid in water and dilute to 1 litre.

A-12.1.2 *Aluminon Reagent* — 0·10 percent aqueous solution. If the solution is reddish, add a drop of ammonia and make it golden yellow.

A-12.1.3 *Thioglycollic Acid* — 10 percent.

A-12.1.4 *Standard Aluminium Solution* — Dissolve 1·757 g of potassium aluminium sulphate in water and make up the volume to 1 litre. Further, dilute 10 ml of this solution to 1 litre. One millilitre of the diluted solution contains 1 μg of aluminium (as Al).

A-12.2 Procedure — Take 50 ml of the sample solution (**A-2.2.1**), acidify with hydrochloric acid, boil and filter. Neutralize the filtrate with ammonia to *p*H 6·8 to 7·2. Add 20 ml of buffer solution, 1 ml of thioglycollic acid solution and 1 ml of aluminon reagent. Heat gently in a water-bath at 100°C for exactly 10 minutes, cool and make up to 100 ml.

For blank, take 50 ml of water and follow the above procedure. Adjust 100 percent transmittance with blank using a photoelectric colorimeter with green 530 nm filter and a 5-cm light path glass cell. Find out the percentage transmittance of the sample and the corresponding optical density and from the standard graph (*see* **A-12.3**), find out the corresponding aluminium content in micrograms.

A-12.3 Take 50 ml of saturated potassium chloride solution in six 250-ml beakers. Use one as a blank. Into the other beakers, pipette out aliquots of standard aluminium solution so as to give 5, 10, 15, 20 and 30 μg of aluminium and make up the volume to 70 ml with water. Add 20 ml of buffer solution, 1 ml of thioglycollic acid solution and 1 ml of aluminon reagent to each beaker. Keep on a water-bath at 100°C for exactly 10 minutes, cool and make up the volume to 100 ml.

Adjust the colorimeter to 100 percent transmittance with the blank on a photoelectric colorimeter using green 530 nm filter and 5 cm light path cell and find out the percentage transmittance of the solution. Find out the corresponding optical densities and plot optical densities against aluminium concentrations.

A-12.4 Calculation

Aluminium content in the sample solution, parts per million $= \dfrac{a}{50}$

Aluminium content in the sample, parts per million $= 3{\cdot}125 \times \dfrac{a}{50}$

where

a = mass in μg of aluminium contained in 50 ml of the sample.

A-13. DETERMINATION OF TITANIUM, COPPER AND NICKLE

A-13.1 Estimation of Titanium

A-13.1.0 *Principle* — Co-precipitation of titanium along with aluminium and colorimetric determination of titanium with tiron (disodium 1, 2-dihydroxybenzene-3, 5-disulphonates).

A-13.1.1 *Reagents*

A-13.1.1.1 *Double boiled distilled water*

A-13.1.1.2 *Aluminium solution* — Same as in **A-11.2.1.3**.

A-13.1.1.3 *Concentrated hydrochloric acid* — *See* IS : 265-1976*.

A-13.1.1.4 *Ammonium solution* — 10 percent.

A-13.1.1.5 *Concentrated sulphuric acid* — *See* IS : 266-1976†.

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*Specification for hydrochloric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

A-13.1.1.6 *Tiron solution* — 10 percent.

A-13.1.1.7 *Thioglycollic acid solution* — 10 percent.

A-13.1.1.8 *Sulphuric acid solution* — 1 : 1 (*v/v*).

A-13.1.1.9 *Standard titanium solution* — Weigh 3·687 5 g of potassium titanium oxalate into a beaker. Add 15 g of ammonium sulphate and 200 ml of concentrated sulphuric acid. Boil for 5 to 10 minutes, cool and make up to 1 litre. Further, dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 5 µg of titanium.

A-13.1.1.10 *Buffer solution* — Dilute 1·6 g of sodium acetate and 60 g of acetic acid to 1 litre with double distilled water.

A-13.1.2 Procedure — Take 2 litres of the sample solution (**A-2.2.1**), proceed in the same manner as for chromium (*see* **A-11.2.2**) and dissolve the aluminium precipitate in 50 ml of boiling concentrated sulphuric acid. Cool and make up to 100 ml, and divide in two parts of 50 ml each. In both, add 10 ml of thioglycollic acid solution. Keep one beaker for the blank. In the other beaker, add 5 ml of tiron solution. Adjust the *p*H of the solution in both the beakers to 3 to 4 by adding ammonia solution. Finally, add 10 ml of buffer solution and dilute to 100 ml with double distilled water. Use the blank to adjust 100 percent transmittance and find out the percentage transmittance of the sample solution on a photoelectric colorimetric using blue 410 nm filter and a cell of 5-cm light path. Find out the corresponding optical density and read the titanium concentration corresponding to the optical density from the standard graph (*see* **A-13.1.3**).

A-13.1.3 *Preparation of Standard Graph* — Pipette out aliquots of standard titanium solution containing 5, 10, 15, 20 and 25 µg of titanium in 250 ml beakers. To each add 15 ml of 1 : 1 sulphuric acid solution, 15 ml of standard aluminium solution and 25 ml of water. Then add 10 ml of thioglycollic acid and 5 ml of tiron solution to each beaker. Adjust the *p*H of the solutions to 3 to 4 with ammonia or sulphuric acid. Finally, add 10 ml of buffer solution and make up the volume to 100 ml. Find out the percentage transmittance using 410 nm blue filter and a glass cell of 5-cm light path. For adjusting 100 percent transmittance, use 50 ml of distilled water treated in the same manner as above. Find out the corresponding optical densities and plot the titanium concentration against the optical densities.

A-13.1.4 *Calculation*

Titanium content in the sample solution, parts per million $= \frac{a}{1\,000}$

$$\text{Titanium content } (X_1) \text{ in the sample, parts per million} = 3{\cdot}125 \times \frac{a}{1\ 000}$$

where

a = mass in µg of titanium contained in 1 litre of the sample.

A-13.2 Estimation of Copper

A-13.2.0 *Principle* — Copper forms a complex with sodium diethyldithiocarbamate solution which is soluble in chloroform giving a yellow coloured solution which is determined colorimetrically.

A-13.2.1 *Reagents*

A-13.2.1.1 *Ammonium citrate solution* — Take 210 ml of concentrated ammonia solution and 150 ml of water in a 500-ml beaker. Add while stirring 200 g of citric acid, cool and make the contents slightly alkaline with ammonia. Add 5 ml of sodium diethyldithiocarbamate solution and extract with 10 ml portions of chloroform, till the chloroform layer becomes water white. Dilute to 500 ml with water.

A-13.2.1.2 *Sodium diethyldithiocarbamate solution* — Take 0·1 g of sodium diethyldithiocarbamate in a 100-ml volumetric flask, dissolve in water and dilute to 100 ml. Filter if not clear and store in a brown bottle. The solution is stable for about a week. It shall be kept in a cool place.

A-13.2.1.3 *Ethylene diamine tetraacetate (EDTA) sodium salt solution* — Dissolve 20 g of ethylene diamine tetraacetate sodium salt in 500 ml of water.

A-13.2.1.4 *Phenolphthalein indicator*

A-13.2.1.5 *Chloroform* — *See* IS : 5296-1979*.

A-13.2.1.6 *Ammonia solution* — 10 percent.

A-13.2.1.7 *Standard copper solution* — Dissolve 0·392 8 g of copper sulphate ($CuSO_4{\cdot}5H_2O$) in a little water. Add 8 ml of concentrated hydrochloric acid and dilute to 1 litre. One millilitre of this solution contains 0·1 mg of copper. Further, dilute 10 ml of the above solution tc 1 000 ml. One millilitre of the diluted solution contains 1 µg of copper.

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*Specification for chloroform, pure and technical (*first revision*).

A-13.2.2 *Procedure* — Take 100 ml of the sample solution (**A-2.2.1**) in a dry separating funnel. Add 5 ml of ammonium citrate solution, 20 ml of EDTA solution and 2 drops of phenolphthalein indicator. Shake vigorously and add ammonia solution till the solution in the funnel turns very slightly pink. Further add 5 ml of sodium diethyldithiocarbamate solution and mix well. Extract the complex with 10 ml portions of chloroform till the chloroform layer becomes water white and make up the volume to 50 ml. Use pure chloroform for 100 percent transmittance adjustment and find out the percentage transmittance of the sample, using a photoelectric colorimeter with blue 560 to 600 nm filter and a 5-ml light path glass cell. Find out the corresponding optical density and from the standard graph (*see* **A-13.2.3**), read the copper concentration.

A-13.2.3 *Preparation of Standard Graph* — Pipette out aliquots of standard copper solution so as to contain 5, 10, 15, 20 and 25 μg of copper. Dilute to 100 ml with water and follow the procedure described for the sample (*see* **A-13.3.2**). Plot the optical densities against the copper concentration on a graph paper.

A-13.2.4 *Calculation*

Copper content in the sample solution, parts per million $= \frac{a}{100}$

Copper content (X_2) in the sample, parts per million $= 3{\cdot}125 \times \frac{a}{100}$

where

a = mass in μg of copper contained in 100 ml of the sample.

A-13.3 Estimation of Nickel

A-13.3.0 *Principle* — Nickel forms a red coloured complex with dimethylglyoxime which is soluble in chloroform and is determined colorimetrically.

A-13.3.1 *Reagents*

A-13.3.1.1 *Sodium citrate solution* — 10 percent.

A-13.3.1.2 *Dilute ammonia solution* — 1 : 20 (*v/v*).

A-13.3.1.3 *Dimethylglyoxime reagent* — 1 percent solution in ethanol.

A-13.3.1.4 *Chloroform* — *See* IS : 5296-1979*.

A-13.3.1.5 *Hydrochloric acid* — 0·5 N.

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*Specification for chloroform, pure and technical (*first revision*).

A-13.3.1.6 *Bromine water*

A-13.3.1.7 *Standard nickel solution* — Dissolve 0·508 8 g of ammonium nickel sulphate [$(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O$] in water and dilute to 1 litre. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 1 μg of nickel.

A-13.3.2 *Procedure* — Take 50 ml of the sample solution (**A-2.2.1**) in a separating funnel, add 5 ml of sodium citrate solution and make the contents slightly alkaline with ammonia solution. Add 2 ml of dimethylglyoxime solution and extract the complex formed with chloroform using 5-ml portions 3 to 4 times.

Collect the chloroform layers in a separating funnel. Add 5 ml of hydrochloric acid solution and shake vigorously. Draw off the chloroform layer and collect the acid layer in a 50-ml volumetric flask. Add 10 drops of bromine water and let stand for a few minutes. Add liquor ammonia till the bromine colour disappears and add 1 ml in excess. Cool to below 30°C, add 1 ml of dimethylglyoxime reagent and dilute to 100 ml with water.

Similarly carry out the blank with 50 ml of water. Adjust 100 percent transmittance on the photoelectric colorimeter with this solution using a green 465 nm filter and a 5-cm light path glass cell. Find out the percentage transmittance of the sample solution and calculate the corresponding optical density. From the standard graph (*see* **A-13.3.3**) read the concentration of nickel.

A-13.3.3 *Preparation of Standard Graph* — Pipette out aliquots of the standard nickel solution so as to give 5, 10, 20, 30 and 50 μg of nickel. Dilute to 50 ml with water and follow the procedure as given for the sample (*see* **A-13.3.2**). Plot the optical densities obtained against the concentration of nickel on a graph paper.

A-13.3.4 *Calculation*

$$\text{Nickel content in the sample solution, parts per million} = \frac{a}{50}$$

$$\text{Nickel content } (X_3) \text{ in the sample, parts per million} = 3{\cdot}125 \times \frac{a}{50}$$

where

a = mass in μg of nickel contained in 50 ml of sample.

A-13.4 Calculation

(Titanium + Copper + Nickel) in the sample, parts per million $= X_1 + X_2 + X_3$.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF POTASSIUM CHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 The samples shall be placed in suitable, clean and dry containers.

B-1.3 The sample containers shall be of such size that they are almost completely filled by the sample.

B-1.4 Each sample container shall be sealed airtight after filling and marked with full details of sampling and the date of sampling.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be selected from the lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* B-2.2)

Lot Size *N* (1)	No. of Containers to be Selected *n* (2)
Up to 50	3
51 „ 100	4
101 „ 150	5
151 „ 300	7
301 and above	10

B-2.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

B-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 For drawing samples, an auger shall be used.

B-3.2 Introduce the auger in different parts of each container to draw an appropriate quantity of the sample from each container.

B-3.3 Out of the portions collected from all the selected containers, an equal quantity of the material shall be taken out and mixed together to form a composite sample of about 250 g. The composite sample so formed shall be divided into three equal parts; one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 The remaining portion of the material from each container shall be divided into three parts and each such separate part shall constitute an individual sample. One set of individual samples (representing the *n* containers selected) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.5 All the individual and composite samples shall be immediately transferred to separate glass bottles and labelled with full identification particulars on the bottles.

B-3.6 The referee samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

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*Methods for random sampling.

B-4. NUMBER OF TESTS

B-4.1 Tests for potassium chloride content shall be conducted on each of the individual samples and tests for all other characteristics given in **3** shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 A lot shall be considered as conforming to the requirements of this specification if the stipulations laid down in **B-5.1.1** and **B-5.1.2** are fulfilled.

B-5.1.1 *For Potassium Chloride* — From the individual test results for potassium chloride content the mean ($\bar{x}$) and the range (R) or mean range ($\bar{R}$) shall be calculated. (Range is the difference between the maximum and minimum of test results.) When the sample size is 10, the test results shall be arranged in two groups of 5 each in the order of their occurrence and the range for each group shall be computed. $\bar{R}$ will be the mean of these two R's. The lot shall be deemed satisfactory for this characteristic if ($\bar{x} - 0{\cdot}6\ \bar{R}$) or ($\bar{x} - 0{\cdot}6\ \bar{R}$) is greater than or equal to the minimum limit specified in Table 1.

B-5.1.2 *For Other Characteristics* — The test results for all other characteristics prescribed in **3** shall satisfy the relevant requirements.

(*Continued on page* 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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